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METAL COATING BY VAPOR DEPOSITION

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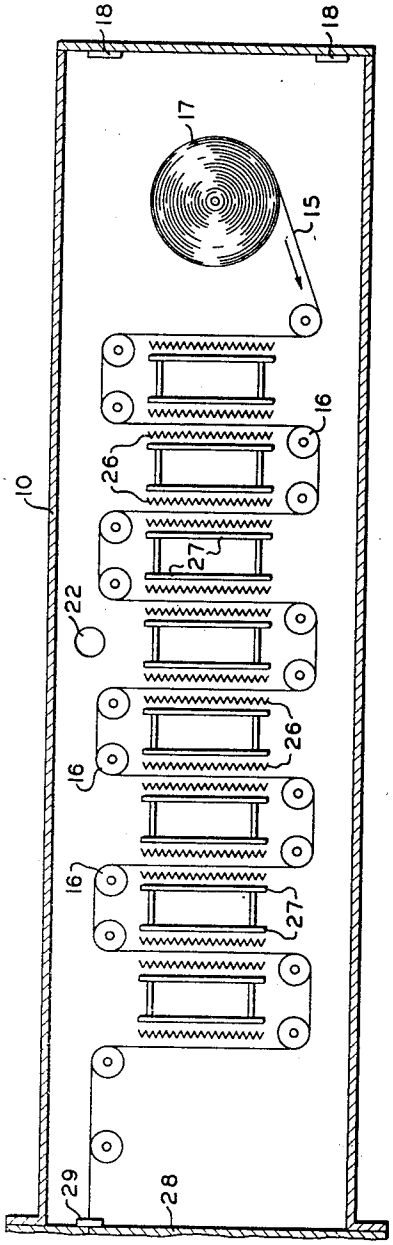


FIG. 1

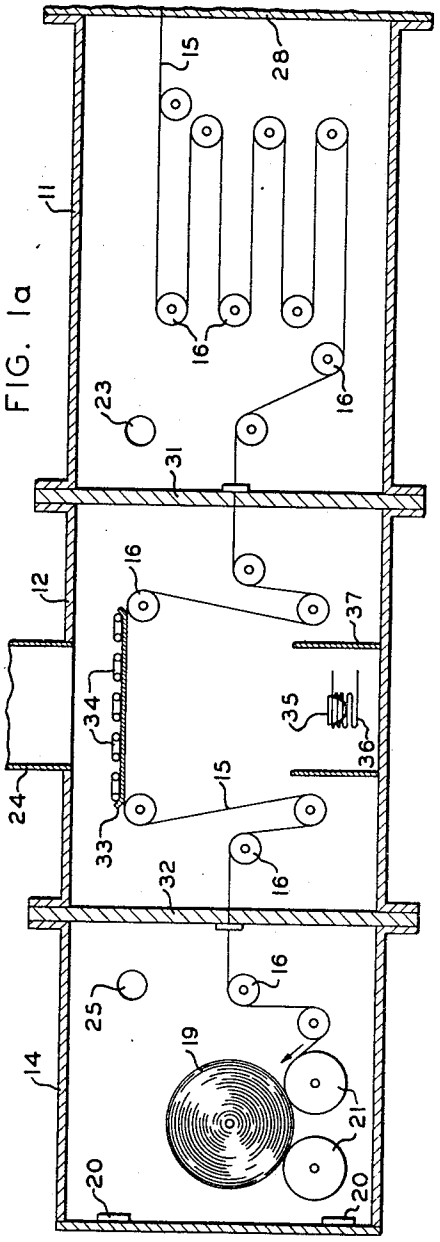


FIG. 1a

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METAL COATING BY VAPOR DEPOSITION

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4 Claims. (Cl. 117—60)

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This invention relates to the deposition on suitable supports or bases of materials, particularly metals, by processes involving thermal evaporation of the material deposited in an attenuated atmosphere and embraces improvements both in method and apparatus. The invention is particularly concerned with the deposition of metals on surfaces or supports containing occluded gases or other volatiles or substances which yield gases or vapors when heated.

The invention is considered especially significant as applied to the coating of cellophane in strip or sheet form with aluminum and it will be described with specific reference to that application, it being understood that the specific description will not be held limitative. By means of the invention an aluminum coating can be applied to cellophane more evenly than heretofore and at a substantially faster rate with fewer operational difficulties.

It has been previously recognized that materials such as cellophane, and other plastic sheeting, sheeting formed of plasticized vinyl chloride or vinyl acetate polymer or vinyl chloride-vinyl acetate copolymer, for example, tend to give off hydrogen, water vapor and possibly other gases and vapors when contacted with a vaporized metal in a vacuum chamber, and that this gas and vapor evolution interferes with the coating operation by rendering it difficult to maintain the vacuum. The hydrogen evolution is especially pronounced in the case of cellophane where aluminum is employed as the coating metal, apparently due to reaction of the aluminum with hydroxyl-containing compounds in the cellophane.

Prior investigators have proposed that prior to the coating operation the surface to be coated be superficially preheated under reduced pressure, it being contended that when this is done, the necessary vacuum in the coating chamber can be maintained without difficulty. It is true that some improvement in results is achieved by so heating the support, but I have not found that the problem is thereby solved.

In accordance with my invention, I substantially cool the support under reduced pressure subsequent to the preheating and before passing it to the coating zone. I have found that when this is done the gas and vapor evolution is still further reduced and markedly so, particularly if the perheating is not limited to the surface to be coated but is extended to the body of the support. In the most preferred form of the invention the support is also subjected to cooling influences during the actual coating thereof.

My invention will be readily understood by reference to the accompanying drawing illustrating schematically, in sectional elevation, a system of apparatus suitable for its practice when the support material is in strip or web form as preferred. In the drawing, Fig. 1*a* represents an extension of Fig. 1.

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The apparatus, as disclosed, will be seen as constituted of four compartments 10, 11, 12 and 14, through which a strip or web to be coated, indicated by the numeral 15, is consecutively passed, the course of the strip through the compartments being determined by rollers or idlers 16. The strip 15 is fed from a feed roll 17 suitably supported within compartment 10. This compartment should be equipped with a pressure tight door for which hinges 18 are indicated. A receiving roll 19 is mounted in the end compartment 14, which should also be provided with a pressure tight door for which hinge elements 20 are indicated, and may be driven in the conventional manner by rotating drums 21 maintained in frictional engagement with the periphery of the roll.

Each of the compartments 10–14 is maintained under a subatmospheric pressure, vacuum lines or conduits 22, 23, 24 and 25 being provided for this purpose. Assuming the material of the strip 15 is cellophane and that the coating metal is aluminum, compartment 12 is best maintained at a pressure not exceeding 0.4 micron of mercury, each of compartments 11 and 14 at a pressure of from 1 to 10 microns and compartment 10 at a pressure of from 50 to 500 microns. A mechanical vacuum pump suffices in the case of compartment 10 and a single diffusion pump is usually sufficient in the case of compartments 11 and 14, but conduit 24, by means of which compartment 12 is evacuated, should connect with a diffusion pump the action of which is supplemented by one or more booster pumps.

In compartment 10, the strip or web 15 is passed over and under heating elements 26 which may be spaced and supported as by insulating members 27. The heating, of course, should not be of an intensity or for a duration causing severe embrittlement of the strip, but the strip should be heated throughout in order that volatiles will be removed from the body as well as from the surfaces thereof. Both the intensity and duration of the heating are dependent on the material of the strip and the limits for any particular material are easily determined by experimentation.

The strip is conveyed from compartment 10 to compartment 11 through a slot in partition 28 therebetween, the slot being covered by a sealing element 29 which substantially prevents equalizing of the pressures in the compartments. Similar elements are secured over slots in partitions 31 and 32 constituting the sides of the coating compartment. These elements may be formed of natural or synthetic rubber or other suitable material of a functionally equivalent nature. Where the material of the strip and the coating opera-

tion are not adversely affected thereby, mercury or other liquid seals may be substituted.

In compartment 11, the heated strip is cooled to reduce the vapor pressure of the volatiles not removed by the heating in compartment 10, the idlers or rollers 16 being so constructed as to enable the circulation of brine, a liquefied gas, or other suitable refrigerant therethrough. Alternatively, the strip may be passed over cooled metal plates, for example. Contact cooling is essential since the amount of air present is inadequate for cooling by convection. The extent of the cooling in the compartment is, of course, determined by the temperature at which the rollers are maintained and the residence period of the strip in the compartment. In general, the more thorough the cooling, the better the results. Thus, in the case of cellophane, every 10° C. drop in temperature reduces the vapor pressure of the contained volatiles 50%. It is my usual practice to lower the temperature of the strip during its residence in the cooling compartment at least 20° C.

From compartment 11, the strip is conveyed to compartment 12, the coating compartment, the strip being there passed under and in contact with a plate 33 preferably of metal, cooled as by coils 34 through which a suitable cooling agent is circulated. Below the plate is positioned a cup 35, shown as heated by a resistance coil 36, and shielded by an annular baffle 37. Cup 35 serves to evaporate the metal deposited on the surface of the strip presented for coating. Where it is desired to coat both surfaces or sides of the strip two cups may be used and certain obvious rearrangements made with respect to the course of the strip through the compartment.

Due to the cooling in compartment 11, the volatiles not removed by the preheating remain substantially dormant, with the result that the necessary vacuum is easily maintained in the coating compartment and the coating operation proceeds smoothly with even deposition of the metal.

The coated strip is conveyed through partition 32, constituting one of the walls of the coating compartment, through seal means as previously described and is wound on roll 19 in compartment 14. In some instances, it may be necessary or desirable to interpose a fifth compartment between compartments 12 and 14. This fifth compartment may represent a spare or additional coating compartment, for example. An additional compartment or compartments may also be interposed between cooling compartment 11 and the coating compartment, it being advantageous at times to apply a preliminary nonmetallic coating before the metal coating is applied. Where possible, however, it is best to carry out the preliminary coating in the cooling compartment.

Exemplary of metals other than aluminum which may be employed in coating operations carried out in accordance with the invention may be mentioned: magnesium, silver, copper, gold, tin, zinc etc. Other supports or base materials in addition to those previously mentioned, which, like cellophane, tend to give off water vapor and/or gases, include paper, textile fabrics, whether constituted of animal, vegetable or mineral fibers, e. g., silk, wool, cotton, rayon, asbestos cloth, fiber glass cloth, etc., artificial leather and the like.

The invention is not limited to the application of metal coatings, being highly useful in the coating of a support with a wax, for example.

I claim:

1. The process of vacuum deposition of a metal on a nonmetallic, flexible substrate which yields gases or vapors when heated, said process comprising the steps of passing said substrate through a heating zone evacuated to a pressure on the order of 50 to 500 microns Hg, heating both sides of said substrate during passage through said heating zone to drive from said substrate occluded gases and other volatiles resulting from said heating, said heating being of sufficient duration and intensity to remove volatiles from the body of said substrate as well as from the surface thereof, immediately thereafter moving said heated substrate from said heating zone to a cooling zone evacuated to a pressure lower than the pressure in said heating zone, providing a vapor barrier between said heating and cooling zones to prevent said evolved gases from entering said cooling zone and condensing on said substrate, cooling said substrate by at least 20° C. to prevent further evolution of gases from said substrate by passing said substrate in contact with at least one cooled surface in said cooling zone, said substrate being moved from said heating zone to said cooling zone and being contacted by said cooled surface prior to the time when the removal of volatiles is sufficient to cause substantial embrittlement of said substrate, passing said cooled substrate from said cooling zone into a coating zone evacuated to a pressure below about 0.5 microns Hg, vaporizing said metal in said coating zone, coating said substrate by condensing thereon said vapors of said metal, and shielding said substrate in said cooling zone from contact with metal vapors generated in said coating zone.

2. The process of vacuum deposition of a metal on a nonmetallic, flexible substrate which yields gases or vapors when heated, said process comprising the steps of passing said substrate through a heating zone evacuated to a pressure on the order of 50 to 500 microns Hg, heating both sides of said substrate during passage through said heating zone to drive from said substrate occluded gases and other volatiles resulting from said heating, said heating being accomplished at a plurality of separate points as said substrate is advanced through said heating zone and being of sufficient duration and intensity to remove volatiles from the body of said substrate as well as from the surface thereof, immediately thereafter moving said heated substrate from said heating zone to a cooling zone evacuated to a pressure lower than the pressure in said heating zone, providing a vapor barrier between said heating and cooling zones to prevent said evolved gases from entering said cooling zone and condensing on said substrate, cooling said substrate by at least 20° C. to prevent further evolution of gases from said substrate by passing said substrate in contact with a plurality of cooled surfaces in said cooling zone, said substrate being moved from said heating zone to said cooling zone and being contacted by said cooled surfaces prior to the time when the removal of volatiles is sufficient to cause substantial embrittlement of said substrate, passing said cooled substrate from said cooling zone into a coating zone evacuated to a pressure below about 0.5 microns Hg, vaporizing said metal in said coating zone, coating said substrate by condensing thereon said vapors of said metal, and shielding said substrate in said cooling zone

from contact with metal vapors generated in said coating zone.

3. The process of vacuum deposition of a metal on a nonmetallic, flexible substrate which yields gases or vapors when heated, said process comprising the steps of passing said substrate through a heating zone evacuated to a pressure on the order of 50 to 500 microns Hg, heating both sides of said substrate during passage through said heating zone to drive from said substrate occluded gases and other volatiles resulting from said heating, said heating being of sufficient duration and intensity to remove volatiles from the body of said substrate as well as from the surface thereof, immediately thereafter moving said heated substrate from said heating zone to a cooling zone evacuated to a pressure lower than the pressure in said heating zone, providing a vapor barrier between said heating and cooling zones to prevent said evolved gases from entering said cooling zone and condensing on said substrate, cooling said substrate by at least 20° C. to prevent further evolution of gases from said substrate by passing said substrate in contact with at least one cooled surface in said cooling zone, said substrate being moved from said heating zone to said cooling zone and being contacted by said cooled surface prior to the time when the removal of volatiles is sufficient to cause substantial embrittlement of said substrate, passing said cooled substrate from said cooling zone into a

coating zone evacuated to a pressure below about 0.5 microns Hg, vaporizing said metal in said coating zone, coating said substrate by condensing thereon said vapors of said metal, shielding said substrate in said cooling zone from contact with metal vapors generated in said coating zone, and additionally cooling said substrate during condensation of said metal vapors thereon by maintaining said substrate in contact with a cooled surface during the time it is exposed to said metal vapors.

4. The process of claim 1 wherein said substrate comprises cellophane and said metal comprises aluminum.

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